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**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of:

Charles W. Wright, et al

SYNTHESIS OF PRIMARY  
HALOSULFONES

Serial No. US 10/712,223

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Examiner: Jean F. Vollano

I hereby certify that this correspondence is being deposited today with the United States Postal Service as first class mail in an envelope addressed to Commissioner for Patents, Washington, D.C. 20231.

Deidra L. Mack  
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November 10, 2004  
Date

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir::

**DECLARATION UNDER RULE 132**

Charles W. Wright of Monroe County, New York, declares as follows:

(1) He is employed as a Senior Development Chemist by Eastman Kodak Co and has been engaged in chemical research for that company since 1991;

(2) Prior to that time he has been employed as a chemist at the Aldrich Chemical Company, Milwaukee, Wisconsin from 1985 to 1991;

(3) His education has included a PhD in Organic Chemistry from the University of Wisconsin in 1991 and a B.S. with High Honors, Chemistry, (ACS certified degree) from the University of Montana in 1980;

(4) Pursuant to his direction and control, as explained in more detail below, experiments were conducted similar to Examples 2 and 3 of Sera U.S. 4,173,481 in an effort to form a bis(vinylsulfonyl) methane

wherein the intermediate bis-halo compound was not isolated as was apparently done between Examples 2 and 3 of Sera.

(5) Using the process of Sera with an alkyl amine or pyridine base without isolation, the results gave an unwanted polymerization corresponding to 54% of the theoretical yield of the intended product.

(6) By comparison, in the present application, examples using bases other than amines such as pyridine or alkyl amine, showed that no significant polymerization occurred.

### **Experimental**

To 16.8 g (0.07233 mol.) of bis(2-hydroxyethylsulfonyl)methane, and 4.23 g (0.053 mol.) of pyridine was added 30 mL (48.9g, 0.411 mol.) of thionyl chloride. When the addition was complete the reaction mixture was warmed to 50° C and stirred for two hours. Vacuum was slowly applied and the excess thionyl chloride was removed to give a solid residue. The nmr of this residue was consistent with the desired bis(2-chloroethylsulfonyl)methane along with aromatic resonance signals consistent with pyridine-containing salt.

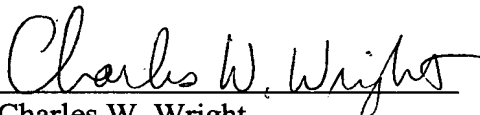
To the non-isolated residue was added 200 mL of acetone and the reaction mixture was stirred to a clear solution. The temperature was set to 21° C. To the solution was added 15 g (~21 mL, 0.148 mol.) triethylamine in a dropwise manner. After the addition was complete, in-process LC showed 68 A% of the desired bis(vinylsulfonyl)methane along with 32 A% of dimerized bis(vinylsulfonyl)methane and further polymerized materials based on the LC retention times of these known peaks. After removal of the amine hydrochloride crystals by filtration, the filtrate was concentrated under reduced pressure to give an oily material that would not crystallize. A sample was prepared for LC chromatography and a significant amount of the sample was not soluble in the acetonitrile solvent, consistent with polymerized material. The LC of the acetonitrile-soluble portion of the sample showed 42.1 A% bis(vinylsulfonyl)methane and 53.7 A% dimerized bis(vinylsulfonyl)methane as well as further polymerized materials. The increase in dimer/polymer for this sample compared with the earlier in-process sample was consistent with a

continuing polymerization reaction. A sample was prepared for nmr spectroscopy and again a significant amount of material was observed to be insoluble in the deuterated acetone solvent. The soluble portion of the sample was consistent with a mixture of the desired bis(vinylsulfonyl)methane, along with traces of pyridine, triethylamine, and other resonances consistent with dimerization and further reaction of bis(vinylsulfonyl)methane. The desired bis(vinylsulfonyl)methane could not be isolated from the polymerized reaction mixture by the normal crystallization solvents and conditions.

### **Conclusion**

The product from this reaction is completely unsuitable for use as a photographic-gelatin hardening agent due to low assay and to the presence of significant amounts of polymeric products. The nonisolation conditions of the bis(2-chloroethylsulfonyl)methane allowed pyridine salts to be present in the subsequent base-sensitive elimination reaction so large amounts of polymeric materials were formed.

The undersigned declares further that all statements made herein of the undersigned's own knowledge are true and all statements made on information and belief are believed to be true. These statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

  
Charles W. Wright

Date: 11/10/04